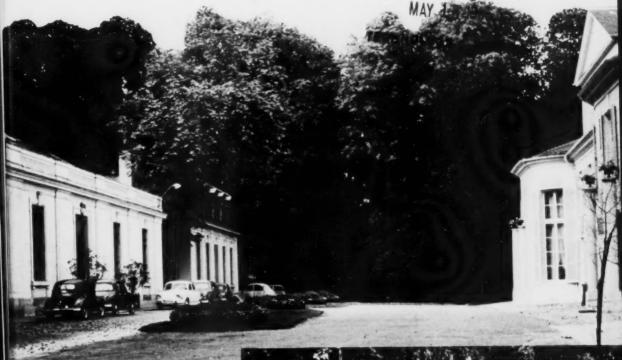
NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

VOLUME 48 . DECEMBER 1964 . NUMBER 12

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12th
International
Conference on
Weights and Measures





U.S. Department of Commerce

A. V. Astin, Director

Luther H. Hodges, Secretary

National Bureau of Standards

NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

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COVER: The International Bureau of Weights and Measures (top), site of the Twelfth General Conference on Weights and Measures, and the new international radiation measurements laboratory (bottom), both at Sèvres, near Paris, France. (See pages 207–208.) tary

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The Twelfth General Conference on Weights and Measures

The Twelfth General Conference on Weights and Measures, held at the International Bureau of Weights and Measures, Paris, France, from October 6 through 13, 1964, was attended by representatives of 37 different nations. Included were leaders from the standardizing laboratories of the major technologically advanced nations. Among the more significant accomplishments of the meeting was the designation of an atomic definition of the second—the international unit of time. This definition is temporarily based on an invariant transition of the cesium atom (see page 209 for further details).

Dr. A. V. Astin, NBS Director, headed the American delegation to the Conference. This delegation included A. G. McNish, Chief of the NBS Metrology Division, and, as advisory members from the State Department, Abraham Friedman, B. C. Gough, K. N. Skoug, Jr., J. A. Bovey, Jr., and Edgar L. Piret.

In other actions taken by the Conference, 12 secondary wavelength standards of length were affirmed. These are as follows:

- A. Wavelengths of krypton 86 $6,458.0720\times10^{-10}$ meter $6,422.8006\times10^{-19}$ meter $5,651.1286\times10^{-10}$ meter $4,503.6162\times10^{-10}$ meter
- B. Wavelengths of mercury 198 5,792.2683×10⁻¹⁰ meter 5,771.1983×10⁻¹⁰ meter 5,462.2705×10⁻¹⁰ meter 4,359.5624×10⁻¹⁰ meter
- C. Wavelengths of cadmium 114 $6,440.2480 \times 10^{-10}$ meter $5,087.2379 \times 10^{-10}$ meter $4,801.2521 \times 10^{-10}$ meter $4,679.4581 \times 10^{-10}$ meter

The values for krypton are within 2×10^{-8} , those for mercury 5×10^{-8} , and those for cadmium 7×10^{-8} of the true values, respectively.

A major objective of the Twelfth General Conference, called 2 years ahead of the regularly scheduled interval of 6 years, was to provide increased financial support for the International Bureau of Weights and Measures. This objective was only partially attained. A special appropriation of about \$300,000 was voted to complete the equipment needs of a new laboratory for

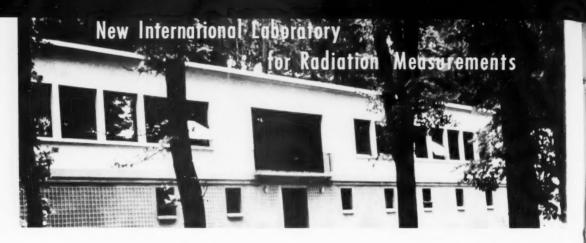
standards for measuring ionizing radiations. However, no firm decision was reached on the proposed increased annual operating budget of the International Bureau. The terms of the Treaty of the Meter require unanimity on budget increases, and some of the delegates (specifically the Soviet bloc nations) had received prior instructions from their governments not to agree to this proposal. It was therefore finally decided that a new budget would be established by mail ballot of all the member nations, and that this would be accomplished by the end of 1964.

The liter, defined up to now as the volume occupied by one kilogram of water, differs from a cubic decimeter by about 28 millionths, and this discrepancy—slightly out of line with other international measurements—has frequently caused difficulty in precision work. The Conference therefore abrogated the old definition, and made the liter merely a special name for the cubic decimeter. The resolution in which this action was taken, however, pointed out that the word "liter" should not be used to express the results of volume measurements of high precision.

In another resolution, the Conference recognized that the curie has been used as the unity of activity of a radioactive substance in a great many countries for a long time, and that in the International System of Units the unit of activity is the second to the power minus one (s⁻¹). It was therefore agreed that the curie should be retained as a special unit, with its assigned disintegration value of $3.7 \times 10^{10} \, \rm s^{-1}$. The symbol formally established for the curie is "Ci."

In other work, the Conference moved forward in approving the research of several of the member nations in extending electrical measurements into the high-frequency region, and in broadening the bases for the International Practical Temperature Scale. During the Conference, the delegates visited the International Bureau's new ionizing radiation laboratory, which had been formally opened the week preceding the Conference (see page 208 for further details).

These secondary standards are the same as those recommended in October 1962 by the International Committee on Weights and Measures. See NBS Tech. News Bull. 47, 29 (1963).



An international radiation measurements laboratory was dedicated September 29, 1964 on the outskirts of Paris, France. This new facility is a significant addition to the International Bureau of Weights and Measures, supported by the member nations that subscribe to the Convention of the Meter.¹ Established as part of this important international scientific organization, the laboratory will promote the use and control of ionizing radiations for medical, industrial, and scientific purposes on a worldwide scale. It will also provide international intercomparisons for radiation measurement standards, including those for x rays, gamma rays, radionuclides, and neutrons.

Dr. Jean Terrien, Director of the International Bureau of Weights and Measures, officiated at the dedication ceremonies. A number of French officials and scientists from all over the world were present for the occasion. Dr. Terrien was assisted by Prof. Richard Vieweg of Germany, President of the International Committee of Weights and Measures, and by NBS Director A. V. Astin, who, as the U.S. representative on the International Committee that governs the International Bureau, had been instrumental in planning the new laboratory and in its realization. Also in attendance from the United States were NBS Associate Director L. S. Taylor, and H. O. Wyckoff, head of the NBS x-ray standards laboratory.

The facility, one of the finest of its kind in the world, is equipped with some of the newest and most accurate equipment available for x-ray, radioactivity, and neutron standards measurements. Space is provided for a high-voltage generator and for various radioactive sources that will be used for calibration operations. The necessary radiation shielding is a built-in feature. André Allisy, an outstanding French physicist, heads a staff of ten scientists who are already at work in their new surroundings.

Since the discovery of nuclear fission over 20 years ago, the use of the ionizing radiations of fissionable materials for the treatment of diseases, for the inspection of industrial products, and as tools for exploring the nature of matter, has steadily increased. In the ensuing years, international intercomparisons of the standards used by many nations to detect, monitor, and regulate the effects of these radiations have been carried out under the auspices of a non-government-

sponsored organization known as the International Commission on Radiological Units and Measurements (ICRU). Dr. Taylor has long been chairman of this organization.

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In October 1958, representatives of several national governments that support the International Bureau of Weights and Measures met in Paris at a session of the International Committee on Weights and Measures. In an action strongly urged by the ICRU, the conferees concluded that the responsibility for international control of radiation standards and measurement techniques should be assumed by the International Bureau. As a consequence, the International Committee appointed a Consultative Committee for the Measurement Standards of Ionizing Radiations, headed by Dr. Astin, to draw up plans for assuming this responsibility.

The first meeting of the new committee was held in April 1959 at the International Bureau of Weights and Measures. Among other recommendations, one was made for the establishment of a new laboratory to carry out the function of providing standards for radiation measurements.

In October 1960, representatives of the member nations attending the Eleventh General Conference on Weights and Measures in Paris approved the recommendation. Negotiations were then undertaken with the French government to obtain property of an extraterritorial character to accommodate the new facility. A parcel of land, adjacent to that occupied by the International Bureau of Weights and Measures (in the beautiful natural environment of Parc de Saint Cloud, overlooking all of Paris) was subsequently donated for this purpose by the French government.

Only limited funds were available at the beginning of the project, so arrangements were made for a Ford Foundation grant (in the amount of \$32,500) to assist in the original planning and design of the laboratory. Additional funds to cover construction and equipment costs were later provided by the member nations.

¹This Convention was signed in 1875 by representatives of 28 nations (since increased to 38). It provided for international agreement in the area of physical measurements and on the bases for the metric system, and at the same time it provided for the establishment of the International Bureau of Weights and Measures.

World Sets Atomic Definition of Time

An atomic definition of the second, the international unit of time, was authorized at 1725 Paris time, October 8, 1964, by the Twelfth General Conference of Weights and Measures, meeting in Paris. The International Committee on Weights and Measures, acting for the Conference, temporarily based the definiion on an invariant transition of the cesium atom, in expectation of a more exact definition in the future. The new definition, which will facilitate the expression of the results of high-precision time and frequency leasurements, is in as close agreement as is experimentally possible with the definition established in 1956, based on the annual orbit of the earth. The 1956 definition has not been formally abandoned. This action was deferred pending the possible replacement of the lemporary atomic definition with a permanent one.

The move toward the redefinition was strongly urged by the American delegation to the international convention. It had previously been proposed by the American, Swiss, and German delegations at the Eleventh General Conference held in Paris 4 years ago.

The action taken increases the accuracy of time measurements to a part in one hundred billion, an accuracy two hundred times greater than that formerly achieved by astronomical means. Moreover, these measurements can be accurately determined in a few minutes, as compared to the many years required to achieve an accuracy only one-hundredth as good by astronomical means.

In the past, the unit of time had been established by astronomers observing the movement of stars across the sky as the earth rotates on its axis. A clock was used to relate the instant of meridian crossing for each individual star to the instant of crossing for other stars. By means of a long series of observations, the rate of the clock could be related to the earth's rotation. The earth itself thus became the timekeeper and the clock was used to interpolate the intervals of time between meridian crossings of different stars.

Pendulum clocks, some of which exhibit a stability of performance to within a few thousandths of a second per day, were employed for this purpose until quartz crystal oscillators having even greater stability were developed for time-interval measurements. Neither of these devices, however, maintains a rate which is as constant as that of the earth.

Prior to 1956 the second was defined as one 86,400th part of the time required for an average rotation of the earth on its axis with respect to the sun. Nevertheless, long before this date astronomers became acutely aware of irregularities in the earth's rotation as comed with the orbital motion of the moon about the earth, the earth about the sun, and various other planetary motions.

detector as Charles Snider pours liquid nitrogen into a cold trap at one end of the instrument. The nitrogen helps form a vacuum

Cesium atomic clock. Roger Beehler adjusts the atomic beam so that cesium atoms can be beamed through the machine without being deflected by molecules of air.

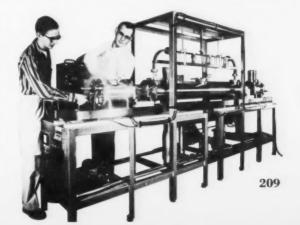
Thus in 1956 an improved arrangement was internationally agreed upon to define the second-called the ephemeris second—as 1/31,556,925,9747 of the time taken by the earth to orbit the sun during the tropical year 1900. Although very exactly stated, this definition could not be realized by astronomical observations with anything like the precision implied by so many digits.

In the 1950's, research on certain atomic transitions indicated that the oscillations associated with them could be realized with great repeatability. One of them, a hyperfine transition in the cesium atom, was related to the ephemeris second with an estimated accuracy of about two parts in a billion. Measurements made with two different instruments, perfected by R. C. Mockler and R. E. Beehler working under J. M. Richardson at the NBS Boulder Laboratories, agreed with each other much more precisely than the measurements made with either instrument could be related to the ephemeris second. This agreement-found to be six parts in 1012, that is, six parts in a million millionmeans that if two clocks are controlled separately by these two instruments, and if there are no other sources of error, the clocks will differ by only one second after running five thousand years.

The exact wording of the action of the Twelfth General Conference is: "The standard to be employed is the transition between the two hyperfine levels F=4, $M_F=0$ and F=3, $M_F=0$ of the fundamental state ²S_{1/2} of the atom of cesium 133 undisturbed by external fields and the value 9 192 631 770 hertz is assigned." This definition is tied up with atomic processes taking place in the cesium 133 atom, the only nonradioactive nuclide of cesium which is different from the radioactive cesium nuclei which are produced in atomic explosions.

How an Atomic "Clock" Operates

Since atomic nuclei and electrons have magnetic moments, they may be lined up parallel or anti-parallel with each other. The energy of one atom may differ from another one, depending upon which alinement prevails. If a transition from one energy state occurs-by reversal of alinement-the atom can emit or



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absorb radiation depending upon the direction of the change. The frequency of this radiation is proportional to the energy difference in the two states as

given by Bohr's relationship.

To realize the frequency of this transition, an atomic beam apparatus is used, commonly called an atomic "clock." Metallic cesium is placed in a small chamber which is heated, causing cesium atoms to be emitted through a small hole into a beam tube. The atoms are separated into two beams by passing through an inhomogeneous magnetic field. Those with parallel magnetic moments go into one beam; those with anti-parallel moments into the other. If now the beams are subjected to an oscillating electromagnetic field, the magnetic moments can be flipped from the parallel to the anti-parallel relationship or vice versa.

Thus when the two beams are passed through a second inhomogeneous magnetic field, parallel alined atoms appear in the beam that was previously all antiparallel, and anti-parallel atoms appear in the other. Only those atoms that have been switched can enter a chamber containing a hot wire where they are ionized and detected. If the frequency of the oscillating field is not exactly that characteristic of the transition, none of the magnetic moments are flipped and no signal is received from the detector. The operator can adjust the frequency of the oscillator which applies the electromagnetic field until he receives a signal. Then the frequency of his oscillator is exactly that of the defined frequency and other oscillators can be adjusted to it.

By well-known electronic techniques the cycles of the oscillator can be counted—9 192 631 770 cycles of the oscillator are equal to exactly one second. In practice, however, oscillators are operated at other frequencies related to the cesium frequency by various circuit devices. These devices are used for the counting; they are operated continuously and they are checked from time to time against the cesium standard.

By use of this technique a very accurate scale of time can be established without reference to the earth's rotation or the planetary motions. Atomic time scales kept for several years at the National Bureau of Standards, under J. A. Barnes, and at the Naval Observatory, under William Markowitz, have shown close agreement with each other and with those kept in several European laboratories, differing by only about a millisecond during the last two years.

The atomic time and frequency are made available to users by broadcast from various radio stations throughout the world, such as NBS Station WWV and

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NBA, operated by the U.S. Navy.

Although the atomic definition of the second enables scientists to maintain more accurate and immediately available scales of time and of time intervals, astronomers are not put out of business in this timekeeping game. The earth's rotation is sufficiently irregular that for the navigator and the space scientist timing signals must be correlated with the earth's rotation. It is still the astronomer's responsibility to tell us when the seasons come and go, when eclipses are to be expected, and when Easter is supposed to be. The new atomic timekeeping is a great aid to the astronomer to help him keep track of the planets. Eventually, he will be faced with the problem of determining whether the time kept by an atomic standard is the same as that kept by the planetary motions.

Attending 53d Meeting of the International Committee of Weights and Measures



First row, left to right: A. V. Astin, U.S.A.; Bourdon, Russia; Terrien, Director of International Bureau; Vieweg, Chairman at time of International Committee of Weights and Measures and former President of PTB-Germany; Sandoval, Mexico; Otero, Spain, Siegbahn, Sweden. Second row: Barrell, Chief of Metrology, NPL England; Howlett, Director, Applied Physics Division, National Research Council, Canada; Interpreter for Russian Delegation; Stulla-Gotz, Austria; Nussberger, Czechoslovakia; Volet, Former Director of International Bureau of Weights and Measures (observer). Third row: Yamauti, Japan; deBoer, Netherlands; Vaisala, Finland, Lehany, Australia. Absent members: Kichlu, India; Marechal, France; Isnardi, Argentina; and Kargatchin, Yugoslavia. (Upon the resignation of Dr. Vieweg, Dr. Howlett was elected Chairman of the Committee on October 13, 1964; Otero, Vice Chairman; and deBoer, Secretary.) Photo courtesy of Comité International des Poids et Mesures.

¹ For additional details on the NBS atomic beam spectrometers, see Atomic frequency standards, NBS Tech. News Bull. 45, 8 (1961).

Solar Flare Ionization

Data analysis by the NBS Central Radio Propagation Laboratory, Boulder, Colo.

Sudden phase anomalies (SPA's) produced by solar flares have been simultaneously observed on several long VLF propagation paths. The observations show that during a solar flare the magnitude of the mean change of reflection height on each sunlit path can be related to the solar zenith angle, and different flares produce different reflection heights. These effects are thought to be related to x rays emanating from the sun.

During most solar flares, the electron density in the lower ionosphere on the sunlit part of the earth is enhanced and ionization is usually produced below the normal D region of the ionosphere. The normal D region is thought to be formed by ionization of the atmospheric constituents by solar Lyman α and cosmic rays; however, this radiation is not adequate to explain the abnormal D-region solar flare effects, such as sudden phase anomalies.

Recent rocket and satellite observations of solar flares have given support to the suggestion that the enhanced D-region ionization during solar flares is due to x rays. The effects of this extra ionization are shown in many ways: the absorption of medium frequency and high frequency signals is increased, the attenuation of VLF and LF atmospherics is often reduced, and the height of reflection of VLF and LF signals is lowered.

Optical observations of the sun have shown that almost all flares follow the same pattern: a rapid rise to peak intensity followed by a short period of peak intensity and a slow return to the condition of preflare brightness. To provide a measure of their relative magnitude, solar astronomers have divided flares into classes of importance according to their area and brightness. In addition to giving an estimate of the peak energy and intensity of the x rays produced by a solar flare, it was hoped that this study i might provide a foundation for classification of solar flares (based on sudden phase anomaly measurements) which would be superior for ionospheric purposes to the optical classifications.

During 1961 the phase and amplitude variations of phase-stabilized VLF transmissions over six long-range propagation paths were observed. This VLF transmission network sampled the variations occurring over approximately one quarter of the earth's ionosphere

and thus provided an excellent means for studying the normal day-to-night variation in ionospheric height as well as the effects of solar flare ionization over a large area of the ionosphere. The phase records for these six transmission paths were examined for sudden phase anomalies (SPA's) during times of known solar flares. In this period some 30 solar flare events occurred, for each of which there were from two to four simultaneous observations of SPA's. Using SPA data obtained from the several VLF propagation paths, the change in effective reflection height and a parameter related to the rate of solar radiation per unit of surface were calculated for each path.

Before proceeding with the interpretation of the data, it was noted that considerable difficulty arose in justifying any particular model for the flare-produced ionization, primarily because the aeronomy of the *D* region is not fully understood and the x-ray energy spectrum of a solar flare is not well known. However, the interpretation of the experimental results in terms of a layer which behaves as a Chapman layer leads to an estimate, which is not inconsistent with satellite data, of the energy and intensity of the x radiation that produced the ionized layer.

¹VIf phase observations of solar flare ionization in the D-region of the ionosphere, by C. J. Chilton, F. K. Steele, and R. B. Norton, J. Geophys. Res. **68**, 5421-5435 (Oct. I, 1963).



VLF transmission network used in studying phase changes accompanying solar flares.

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Thermal Degradation of Polyisobutylene

Isobutylene is the main constituent of a synthetic rubber widely used by the tire industry. A better understanding of its thermal stability could lead to additional uses as well as to improved methods of copolymerization with other materials.

The thermal degradation of both high- and low-molecular-weight polyisobutylene has recently been investigated. Results of the study indicate that the polymer chain ruptures randomly at carbon-carbon bonds along the chain backbone, and that the rate of degradation has little dependence on molecular weight from about 30,000 to 5,000,000.

In a previous study,² ethylene and propylene polymers were found to be more stable than polyisobutylene. The present work was therefore undertaken to obtain detailed data on polyisobutylene and to deduce the decomposition mechanism from molecular weight changes during degradation.

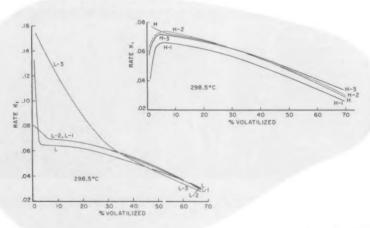
The experiments were carried out using fractionated and unfractionated commercially prepared high-purity polyisobutylenes of high and low molecular weights. Samples weighing from 4 to 5 mg were pyrolyzed in a vacuum at 298.5 °C for about 2 days. The temperature was kept constant to within ± 0.2 °C by means of an electronically controlled thermostat. The sample was held in an electronic microbalance which automatically recorded the amount of volatilization during a run.

Before pyrolysis, samples were each distilled in 1 percent benzene solutions and fractionated stepwise with distilled acetone as the precipitant. Molecular weights of the fractions were determined from measurements of viscosity in iso-octane at 40 °C. The whole high polymer (molecular weight 1,800,000) (H) gave fractions of 4,800,000 (H-1), 1,980,000 (H-2), and 700,000 (H-3) molecular weight. The whole low polymer (molecular weight 49,000) (L) gave fractions of 234,000 (L-1), 44,200 (L-2), and 24,500 (L-3) molecular weight.

The similarity of the rate curves obtained for both the high and low polymers shows that molecular weight has little effect on thermal degradation. Extrapolation of the curves to the rate axis after only 4 percent volatilization yields values of about 0.07 percent per minute with a minimum of deviation except in the case of the lowest fraction of the low polymer (L-3). The high initial rate of this lowest fraction probably indicates the presence of more low molecular weight material than might have been expected from the fractionation.

Five residues obtained from pyrolysis of the middle fraction (H–2) of the high polymer were pyrolyzed further at $300~^{\circ}\mathrm{C}$ for periods ranging from 24 to 660 min. At the end of each run, the sample's molecular weight was determined by both viscosity and osmotic pressure methods. The results showed a rapid drop in molecular weight from 1,980,000 to 95,000 with only

(Continued opposite page)



Left: Curves show percent of sample volatilized per minute (ordinate), as a function of the total amount volatilized (abscissa). Right: Similar curves for polyisobutylene samples with lower molecular weights.

Investigations by Donald McIntyre, James H. O'Mara, and Sidney Straus of the NBS Institute for Materials Research

New Look at Earth's History

Geophysicists may have to re-examine their beliefs concerning the history of the planet Earth as a result of an experiment recently conducted at the Bureau. Measurements of the rates of helium ion reaction with oxygen and nitrogen molecules indicate the possibility that events may have occurred in the past few million years which boiled off a portion of the earth's atmosphere.

In the simplest terms, the experiment was performed by adding oxygen (or nitrogen) to rapidly flowing, weakly ionized helium gas and detecting the ion species downstream by means of a mass spectrometer. The results showed that helium ions are destroyed somewhat more readily with nitrogen molecules than with oxygen molecules. This result is in contrast to general belief among geophysicists based on theoretical expectations and has several important geophysical

consequences.

Helium ions occur in the earth's high atmosphere due to ionization of neutral helium by extreme solar ultraviolet radiation. If the loss of the helium ions were through reaction with oxygen molecules as previously assumed, the neutral helium atoms resulting could have sufficient energy to escape from the earth. This mechanism might have explained a steady-state helium terrestrial atmosphere. Helium ion loss in reaction with nitrogen, now known to dominate, does not contain this escape possibility, so that geophysicists must find another escape mechanism or abandon the steady-state theory.

Experiment directed by E. E. Ferguson of the Atmospheric Collision Processes Laboratory in the NBS Central Radio Propagation Laboratory

The basic problem is that the amount of neutral helium present in the earth's atmosphere would have been produced in only a few million years by radioactive decay of uranium and thorium in the earth's crust, whereas the age of the earth is a few billion years. As a result, says Dr. Ferguson, "we may be forced to conclude that events occurred in the past few million years which boiled off the earth's helium atmosphere and that the helium concentration is not steady state and is now increasing with time."

Another important consequence of the laboratorymeasured helium ion loss rate with nitrogen is that it places one of the most severe constraints to date upon certain atmospheric parameters, principally the extreme solar ultraviolet radiation intensity, the neutral nitrogen density, and the helium ion density at very high altitudes. The concentration of helium ions has been measured by rocket-borne mass spectrometers, as has the extreme solar ultraviolet radiation, so that the nitrogen concentration may be the least well-known quantity. Analysis of this problem has only begun, and only very tentative conclusions can now be made, but it appears that the laboratory results may support the theory of a lower nitrogen concentration in the earth's very high atmosphere than has been ordinarily assumed.

1.5 percent volatilization. At 10 percent volatilization, the molecular weight dropped to 28,000 and then tapered off to about 15,000 at about 50 percent volatilization. The rapid initial drop strikingly demonstrates that the polymer degrades predominantly at random points along the polymer chain and not at the chain ends.

A comparison of the polyisobutylene rate curves with those found earlier (see footnote 2) for polyculylene and polypropylene shows that polyisobutylene decomposes about 15 times faster than polypropylene and about 600 times faster than polyethylene. The high polyisobutylene rate is probably due to its easy disproportionation and to radical formation. The small "zip" length of the polymer chain and the intermolecular and intramolecular transfer of hydrogen

atoms soon after the initial random breaks may account for the lack of a broad maximum in polyisobutylene degradation. In some earlier experiments, only 20 percent of the monomer was distilled off, which lends support to the theory of the transfer reactions.

Pyrolysis of polyolefins, by L. A. Wall and S. Straus,

J. Polymer Sci. 44, 313 (1960).

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¹ For details, see Thermal degradation of fractionated high and low molecular weight polyisobutylene, by D. McIntyre, J. H. O'Mara, and S. Straus, J. Res. NBS 68A (Phys. & Chem.), 153 (1964).

^aPyrolysis of polyisobutene, polyisoprene, polybutadiene, GR-S, and polyethylene in a high vacuum, by S. L. Madorsky, S. Straus, D. Thompson, and L. Williamson, J. Res. NBS 42, 499 (1949) RP1989; and J. Polymer Sci. 4, 639 (1949).



Standard Materials

Revised Microcopy Resolution Test Charts

Starting December 1, 1964, newly revised test charts ¹ for evaluating the resolving power of microfilming systems are being issued by the NBS Institute for Basic Standards in place of those previously available. A slight alteration in the layout of the spatial frequency patterns on the chart permits the frequency range to be extended to higher or lower frequencies. In addition, small changes in the numbers assigned to these patterns make them conform to international usage. Government specifications for microfilming services have been amended to permit the use of either the old or the new charts in determining resolution.

American industry and Government spend about a third of a billion dollars annually to microfilm records and preserve films. To assure that the microfilmed images are of adequate quality to store the required information, microfilm contracts generally stipulate that the resolving power of the complete microfilming system being employed is to be evaluated by means of NBS microcopy resolution test charts.

To make these evaluations, the resolving power of the system is measured under actual microfilming conditions. First, the charts are placed in several locations on the camera copy board and are photographed. The resulting image is then examined with a microscope. The number of lines per millimeter in the smallest spatial frequency pattern in which the lines can be counted with certainty gives the resolving power of the system.

The charts containing these patterns have been produced by the NBS photographic research laboratory for over 20 years, as part of the Bureau's standard materials program.² In recent years, however, the widespread use of photographic techniques in technological applications—for example, in the manufacture of microminiature electronic components—has greatly increased the demand for these charts. Over 16,000

have been issued thus far this year. Together with the increasing demand, a need has developed for more versatile charts having a wider range of spatial frequencies. The new charts were therefore designed to meet this need.

Produced photographically by contact printing on glossy paper, the charts contain 21 frequency patterns (as do the old charts) for measuring resolving power of from 1 to 10 lines per millimeter. The old chart is shown on the left in the illustration and the new one, identified by the 1963 date of its design, is shown on the right. To achieve greater economy and efficiency in production, six charts (each $31/3 \times 4$ in.) instead of four (dimensions of the old chart are 4×5 in.) are printed on sheets of 8×10 -in. paper. The center of the array of patterns on the new chart is in the horizontal lines of the finest pattern.

In the rearrangement of the frequency patterns, space is available in the center of the array for a tenfold reduction of the chart so that the range of frequencies can be extended to 100 lines per millimeter. Similarly, to extend the range to lower frequencies, a standard chart may be mounted in the center of a tenfold enlargement. In the new arrangement, a square array of patterns with frequencies ranging from 2 to 10 lines per millimeter is provided for an abridged version of the chart, if desired.

To assure the continuity of microcopying procurement programs, certain characteristics of the old chart were retained in the new design. These include chart color, line type, contrast, line-to-space ratio, number of lines per pattern, and length-to-width ratio, of lines. No significant difference in measurements will therefore result in changing from the old chart to the new.

The resolution of an entire photographic system may, at some points in the field, differ materially in orthogonal directions. To facilitate the measurement of this

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characteristic, the orthogonal arrangement of the two groups of pattern lines—one group vertical, the other horizontal, and both parallel with the chart edges—was retained. As in the old chart, the selected frequencies are in a geometrical series to provide proportional frequency increases over the entire range from the largest to the smallest pattern.

The numbers assigned to the frequency patterns of the new chart are based on the "preferred-number" series contained in the International Organization for Standardization Recommendation R3, adopted at the 1953 meeting of the organization in Geneva, Switzerland. With the exception of number 1.25 (indicating a resolution of 1.25 lines per millimeter), all the numbers are rounded to two digits. The change from number 3.5 on the old chart to 3.6 on the new one represents the greatest percentage change, namely 2.86 percent. Since observers often disagree, however, on the reading of patterns differing in frequency by 12.5 percent, this change may be regarded as practically imperceptible.

The proposed revisions were evaluated in advance by a number of users so that changes could be made, if necessary. The new design, however, proved to be satisfactory.⁴ The NBS Microcopy Resolution Test Chart has higher frequencies than the chart accompanying NBS Circular 533.5 The latter chart is designed to measure the resolving power of camera lenses—including those used for photogrammetric purposes—and telescopes and binoculars.

¹ The new chart, designated Microcopy Resolution Test Chart—Standard Sample 1010, may be obtained from the Standard Samples Unit, National Bureau of Standards, Washington, D.C., 20234, at 20 cents each on a minimum order of \$1.00 (stamps not accepted).

minimum order of \$1.00 (stamps not accepted).

*Standard materials issued by the National Bureau of Standards are described in NBS Misc. Publ. 241, Standard Materials, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (30 cents). Up-to-date supplementary inserts, which are issued periodically, are available upon request directly from the National Bureau of Standards.

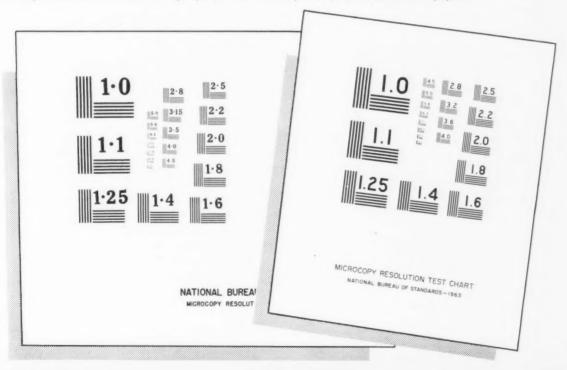
request directly from the National Bureau of Standards.

This recommendation may be obtained from the American Standards Association, 10 East 40th Street, New York, N.Y., 10016.

⁴The NBS microcopy resolution test charts, by B. H. Fouquet, Proceedings of the Twelfth Annual Meeting of the National Microfilm Association, pp. 67-76 (1963).

^a Method for determining the resolving power of photographic lenses, by F. E. Washer and I. C. Gardner, NBS Circ. 533, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (§1.75).

Left: NBS Microcopy Resolution Test Chart in use from 1941 to 1964. (The size of the legend was changed during this interval.) Right: The NBS Microcopy Resolution Test Chart which was designed and announced in 1963 and went on sale December 1, 1964. Although the charts are shown actual size, they are for illustration only and should not be used for test purposes.



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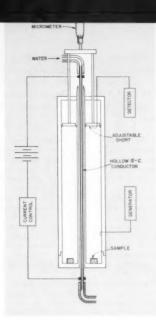
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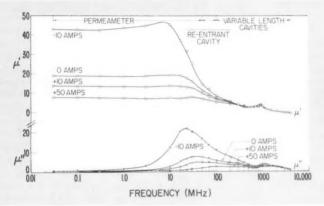
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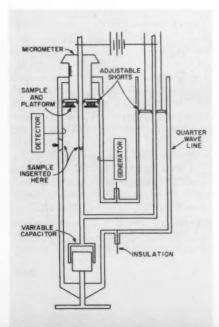
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Reversible Magnetic Permeability



Technique devised by Cletus A. Hoer, Robert D. Harrington, and Alvin A. Rasmussen of the NBS Institute for Basic Standards, Boulder (Colo.) Laboratories



Top of page: Modified half-wavelength cavity opens to permit the sample of magnetic material to be put in place. Bottom of page: Quarter-wavelength tuned stub cavity. One connection for the d-circuit is at the shorted end of the stub; the two magnetizing circuits are separated by insulation between the mating flanges of the main cavity and the stub. Center: Plots of permeability of a magnesium ferrite-Each graph of $\mu^{\prime\prime}$, the loss of the sample, rises from zero at both high and low frequencies. The sample was cycled at each frequency several times at $\pm\,100$ A, after which measurements were made as the current was decreased from $\pm\,100$ A to zero, reversed, and increased to $\pm\,100$ A.

NBS Technical News Bulletin

Instruments used for making initial permeability measurements at the Bureau have been modified to make parallel reversible permeability measurements.\(^1\) An rf permeameter previously developed at the Bureau is used at frequencies up to 50 MHz. For higher frequencies to 100 MHz, a variable-length re-entrant cavity with a quarter wavelength line is used. Two variable-length half-wave cavities were modified for measurements of reversible magnetic permeability at frequencies from 100 MHz to 3 GHz.

The instruments are used in measuring the reversible permeability and loss as a function of the d-c field at a fixed frequency. A complete set of data obtained at many frequencies enabled a graph to be made of μ' (permeability) and μ'' (loss) versus frequency for different d-c current levels.

Modification of the rf permeameter. The radiofrequency permeameter previously developed at the Bureau for permeability measurements up to 50 MHz consists of a toroidal primary winding and a secondary winding formed by a shorting enclosure, one end of which is removed for the insertion of a toroid of the material being tested.² The d-c winding added to form the modification consists of a single turn, in the form of a central stem and coaxial outer shell, enclosing only the test core. This arrangement so limits coupling between the rf and d-c circuits that rf filters are needed in the d-c circuitry only above 4 MHz.

Reversible permeability is measured in the same way as is initial permeability, except for application of the d-c field. A recent study of the permeameter equations for evaluating p' and μ'' has resulted in a set of exact equations which are independent of the transformer characteristics. This permits the permeameter to be evaluated at a given frequency with a standard sample of known μ' and μ'' . The permeameter is calibrated in operation by measurements of its input impedance with the chamber open, closed, and then containing the standard sample.

Re-entrant cavity as a permeameter. A variable-length re-entrant cavity with a quarter wavelength line was developed for reversible permeability measurements at frequencies between 50 and 100 MHz. The complex permeability is determined from the change in the cavity resonance length with insertion of the

sample. No capacity calibration need be made and no expensive frequency-measuring equipment is needed. This method also minimizes errors introduced by supports and discontinuities in the line.

Direct current passing through the center conductor of the cavity provides a circumferential field in the vicinity of the sample mounted on the tuning plunger. A thin sheet of insulation between the flange of the quarter wavelength line and the cavity confines the direct current to the center conductor.

Tuned half wavelength cavity. The use of tuned variable-length cavities for initial permeability measurements above 100 MHz suggested to Institute scientists that the same cavities might be useful for reversible permeability measurements if the center rf conductor were hollow to contain an insulated d-c conductor. Two such instruments were designed and tested, one at frequencies below 300 MHz and a smaller one for precise measurements above that frequency.

Several precautions were taken in the design and use of both experimental cavities. They are used in the vertical position to obtain a straight center conductor without any supports. The center conductor and shorting plate of the smaller cavity were constructed integrally to avoid any discontinuities near the sample at high frequencies. This arrangement gives a uniform distribution of rf current around the center conductor where it joins the shorting plate, and hence a uniform rf field surrounding the sample. The entire sliding-short-and-center-conductor assembly is removable for positioning the sample on the shorting plate. The temperature of both cavities is fixed, for maximum accuracy, by circulating temperature-stabilized water through the hollow d-c conductor.

The data obtained by measuring the reversible permeability and loss as a function of the d-c field at each frequency enable a set of curves to be drawn to relate permeability with d-c field and frequency. These curves enable the spectrum to be obtained for any desired current.

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¹ Parallel reversible permeability measurement techniques from 50 kc/s to 3 Gc/s, by Cletus A. Hoer and R. D. Harrington, J. Res. NBS 67C (Eng. & Instr.). 259-265 (July-Sept. 1963).

² Advances in the design and application of the radiofrequency permeameter, by A. L. Rasmussen, A. W. Enfield, and A. Hess, J. Res. NBS 56, 261-268 (May 1956).



In addition to its thermocouple calibration services, the NBS Institute for Basic Standards conducts research to identify and characterize pairs of alloys useful for reliable thermocouple temperature measurement. Some highlights of this research on thermocouple materials are described below.

Tungsten versus Rhenium. Various investigators have found that thermocouples made of tungsten (W) versus rhenium (Re) have the advantages of high thermoelectric output, high melting point, chemical stability in non-oxidizing surroundings, low cost of the tungsten element, and good reproducibility after thermal cycling to 2200 °C. Some disadvantages are the high cost of Re, brittleness of W, and erosion of W by the water cycle effect. In "Studies on the tungsten-rhenium thermocouples to 2000 °C," J. Res. NBS 67C (Eng. & Instr.), No. 4, 337–352 (Oct.–Dec. 1963), D. B. Thomas describes experiments with different lots of W and Re wire obtained from various domestic sources.

Eleven thermocouples were made from the available wire and tested at 100 °C intervals from 0 to 2000 °C. Measurements were made in a helium-filled tantalum tube furnace 2 using a Pt-1 percent Re versus Pt-30 percent Re thermocouple as the standard instrument for temperature measurements to 1000 °C and an optical pyrometer for measurements from 1000 to 2000 °C. From the experimental data, tables of emf versus temperature at 5 °C and 10 °F intervals were calculated by computer. With the materials included in this study, a maximum thermoelectric power of 18.33 µV per deg C occurs at 715 °C. The tests show that the emf produced by a particular thermocouple is highly dependent on the lot of wire used and on the annealing that the thermocouple received before use. The emfs of the 11 thermocouples had a maximum spread at 2000 °C of 263 µV, equivalent to ~44 °C.

Platinum versus Platinum-Rhodium. Platinum versus platinum-rhodium thermocouples have long been used for measurements to about 1700 °C. In fact, the International Practical Temperature Scale is currently defined over the range 630.5 to 1063 °C in terms of a platinum versus platinum-10 percent rhodium thermocouple. By using Pt-Rh combinations for both legs of a thermocouple, improved stability, higher operating temperatures, and increased mechanical strength can be achieved. Of the many possible combinations, the Pt-6 percent Rh versus Pt-30 percent Rh thermocouple seems to be the best suited for further development and standardization. (Considerable work has been done on this combination in Germany.)

Acting on a request made by the ASTM, the Bureau obtained samples of wires from three American manufacturers and conducted an extensive series of measurements on 11 thermocouples made from these wires. A preliminary emf-temperature table has been prepared that agrees within the experimental error with a similar table produced in Germany, and this is currently being circulated to other laboratories for evaluation before publication. This work has been described by George Burns in a talk given at the Sept. 23–27, 1963, meeting of the SAE, Los Angeles, Calif., "Studies at NBS of the platinum—6 percent rhodium versus platinum—30 percent rhodium thermocouple—A preliminary report" (paper 750B).

Iridium-Rhodium versus Iridium. As part of a continuing investigation of thermocouple materials, G. F. Blackburn and F. R. Caldwell published "Reference tables for thermocouples of iridium-rhodium alloys versus iridium," J. Res. NBS 68C (Eng. & Instr.), No. 1, 41-59 (Jan.-Mar. 1964). Reference tables from 0 to 2150 °C for 60 percent Ir-40 percent Rh and 50 percent Rh-50 percent Ir versus Ir are presented in this paper; limited (100 deg F intervals) tables for Ir-10, -25, -75, and -90 percent Rh versus Ir are also included. From an analysis of the data it appears that the 50 percent Ir-50 percent Rh versus Ir gives about the maximum emf—12.2 mV at 2150 °C and may be the optimum combination for thermocouples of this type. Thermocouples of nominal composition of 40, 50, or 60 percent Rh versus Ir can be used for measuring temperatures to 3900 °F to an accuracy of ±40 °F with the appropriate tables. Calibration of individual thermocouples will of course permit greater accuracy.

Reference tables for 40 percent Ir-60 percent Rh versus Ir were published in J. Res. NBS **66C** (Eng. & Instr.), No. 1, 1-12 (Jan.-Mar. 1962). A general survey of thermocouple materials for use above 0 ° C is available in NBS Mono. 40, *Thermocouple Materials*, by F. R. Caldwell.³

See p. 77 of Calibration and Test Services, NBS Misc. Publ. 250, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. Price 70 cents. This document describes and lists prices for all NBS calibration and test retrieves.

² A furnace for thermocouple calibrations to 2200 °C, D, B, Thomas, J. Res. NBS 66C (Eng. & Instr.), No. 3 (July–Sept. 1963).

[&]quot;Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. Price 30 cents.

Ceramics Used for Transducer Components

The use of ceramic materials for insulating sections has extended the application of accelerometers and shakers to higher frequencies. The NBS vibration measurement laboratory has reported the successful use of alumina disks as accelerometer bases and as shaker bases and top sections. Miniature accelerometers using this material weigh only about a gram and have resonance frequencies of 150 to 200 kHz, and shakers using it have performed satisfactorily to higher frequencies than conventional ones.

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The increasing use of vibration tests in predicting component failure by simulating extended use has led the Bureau to seek improvements in present techniques and devices. Many accelerometers and shakers are piezoelectric devices; each of these consists of a block of piezoelectric material attached to a mounting base on one side and to a top section on the other. In the accelerometers, changes in tension or compression of the piezoelectric element generate electrical potentials across it, to which connection is made by opposed contacts. Piezoelectric shaker tables use the reverse process to produce mechanical vibration from an electrical signal.

Metal has been used for bases and top sections of conventional piezoelectric shakers and compression-type accelerometers because of its high density and mechanical strength. This sometimes simplifies making electrical connections, but it also makes electrical isolation difficult.

The Bureau's transducer research has shown that alumina has excellent qualities of electrical insulation, stiffness, and density and is therefore well suited for use in transducer components. One alumina found to

Right: Miniature piezoelectric accelerometers are assembled on this fine-positioning press, which holds the piezoelectric element above the base while the 0.003-in. (about 0.1-mm) layer of epoxy between hardens. An alumina ceramic base is in place on the anvil of the press. Another is on its side in front of it, to the left of smaller piezoelectric assembly.

Below: Earle Jones attaches a ceramic-base piezoelectric accelerometer to a shaker by means of a layer of adhesive.



Technique reported by Earle Jones of the NBS Institute for Basic Standards

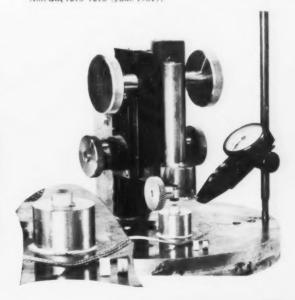
be particularly satisfactory has a Young's modulus of about $345 \times 10^9 \, \mathrm{N/m^2}$, a hardness of 80 Rockwell 45N, and a specific gravity of 3.80 to 3.90. These characteristics—stiffness, hardness, low density, and electrical insulation—make such materials strongly competitive with metals for transducer applications.

Ceramic base sections have now been used at the Bureau in miniature accelerometers having extremely high unmounted resonance frequencies. Typical devices weigh about a gram, resonate at 150 to 200 kHz, and are secured to the vibrating surface being studied by means of an adhesive.

The piezoelectric shaker tables using ceramic bases and top sections have given extremely satisfactory results. They produce a larger vibration amplitude, for the same driving power, free of resonance mode breakup to higher frequencies, than do shakers using metal parts. The results obtained with the use of ceramic bases and top sections for piezoelectric devices have led to their use in moving coil transducers also.

The success of ceramic accelerometer and shaker components has been due in part to the use of epoxy cements, unknown when early transducers were made, for bonding ceramic to other materials. Modern technology has provided adhesives for mounting the accelerometer also, eliminating the weight and undesirable effect on resonance frequency of other means of mounting.

¹Letter to the Editor, by E. Jones, J. Acoust. Soc. Am. 36, 1215-1216 (June 1964).



December 1964

Granular Content of SULFUR MORTARS

Method devised by C. L. Thompson, Research Associate of the Cement and Concrete Reference Laboratory, at the NBS Institute for Applied Technology

A new method ¹ of determining granular content of sulfur mortars requires less time and equipment than the recommended ASTM test method. It should be useful to concrete-testing laboratories that do not have the equipment necessary to perform ASTM test method C 287.

Sulfur mortar has come to be widely used in the last decade as a capping material for concrete specimens. Sulfur mortar capping permits the test specimen to be loaded uniformly by furnishing flat bearing surfaces.

In many instances after testing is completed the sulfur mortar is struck off, remelted, and reused. The resultant melting and remelting of the mortar, however, drives off some of the sulfur, thereby changing its physical properties. The mortar should therefore be tested for its granular content at frequent intervals to determine its condition.

The ASTM test method (C 287), in which the sulfur content is extracted by dissolving it in carbon disulfide, is not only tedious, but also requires special apparatus that many of the concrete-testing laboratories do not have. Therefore, a simple yet relatively accurate test method involving less time and equipment is desirable.

In the NBS method, the cap is removed from the cylinder and crushed. A 20-g sample is placed in a previously ignited, cooled, and weighed crucible. The crucible is heated over a Bunsen burner adjusted so that the sulfur is burned off without splattering. When the sulfur is completely consumed, the residue is ignited at a higher temperature for 30 min. Then the crucible and residue are cooled in a desiccator and weighed. The ignition, cooling, and weighing phases are repeated until a constant weight is obtained. This constant weight is considered to be the weight of the granular materials. The test must be done under a hood or in a well-ventilated area, as considerable sulfur dioxide is generated during the test.

The amount of granular material can be determined to the nearest 0.01 g, an accuracy greater than that needed for normal applications. This relatively accurate and simple method should therefore be very useful to concrete-testing laboratories.

Changes in NBS Radio Broadcasts

Closer Agreement of NBS and Navy Epochs

In accordance with the policy of cooperation between NBS and the U.S. Naval Observatory, changes in the time ticks broadcast by stations of both were made to improve their agreement. The ticks broadcast by NBS stations WWV (Greenbelt, Md.), WWVH (Maui, Hawaii), and WWVB (Fort Collins, Colo.) were retarded 1.0 milliseconds at 0000 UT on 1 October 1964. At the same time the ticks broadcast by Navy stations were advanced 1.6 milliseconds. This brings into closer agreement epochs of the time standards broadcast by the United States Government.

North Pacific Propagation Forecasts

Station WWVH no longer gives forecasts of propagation in the North Pacific, effective 0000 UT on 1 November 1964. These propagation forecasts were formerly given hourly in Morse code, during the first

half of the fifth minute of each hour. The predictions were supplied by the NBS North Pacific Radio Warning Service at Anchorage, Alaska, which is reorienting its mission and will be known as the CRPL High Latitude Space Environment Monitoring Station. It will concentrate on the monitoring and rapid reporting of geophysical events of interest not only to HF radio propagation users, but also to those interested in changes in our space environment. Thus, though HF propagation notices will no longer be given on WWVH, it is anticipated that at a later date, as the need develops, some other statement upon solar and geophysical activity will be disseminated in their place.

Propagation forecasts will continue to be given every five minutes from station WWV. Although the WWV forecasts are for the North Atlantic area, predictions of disturbed conditions throughout the northern auroral region will be generally applicable to the North

Pacific area also.

¹ For details, see A simplified method for determining the amount of granular materials in sulphur mortars. by C. L. Thompson, ASTM Mater. Res. & Stds. (in press).

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